Catalytic Analysis. XX : Microdetermination of Selenium with Methylene Blue and Sodium Sulfide

Catalytic Analysis. XX
Microdetermination of Selenium with Methylene Blue and Sodium Sulfide*

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Synopsis

Applying the catalytic behavior of selenium to the reaction between Methylene Blue and sodium sulfide, the method for the determination of microamount of selenium was studied. Using 3.5 ml of sodium sulfide solution and 0.4 ml of Methylene Blue solution (0.025%), 2～14 γ of selenium at 20°C and 5～50 γ of selenium at 15°C were determined in 10 ml solution.

I. Introduction

It has already been reported[1] that selenium acts as catalyst for the reaction between sodium sulfide and Methylene Blue, and its behavior has been utilized as a sensitive test for selenium. In the present case, the relation between the decoloration velocity of Methylene Blue and the amount of selenium was studied and the determination of microamount of selenium was investigated.

II. Experiments and results

1. Apparatus and reagents

Reagents used were as follows: sodium sulfide was 0.1550 N aqueous solution (the concentration was determined by the iodometry). Selenium solution was an aqueous solution of selenous acid (Se 0.1 mg/ml). The content of selenium in the solution was determined by weighing it as metallic selenium reduced with sulfur dioxide gas, and the solution was diluted until it became a suitable concentration for the experiments when used. Methylene Blue solution was aqueous solution (1 per cent) and also diluted when used. During experiments, reagents were all kept at constant temperature using thermostat.

2. Experimental method

In 50 ml Erlenmeyer flask a suitable volume of water was added until the total volume became 10 ml and then sodium sulfide solution, selenous acid solution and Methylene Blue solution were added in the order named. The flask was shaken well and then the solution was transferred to a test tube and the time required for the decoloration was measured at 5°, 15° and 20°C.

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1) F. Feigl, P. West, Anal. Chem., **19** (1947), 351.

3. Experiments

(i) Amount of N/10 sodium sulfide solution

Using 0.4 ml of 0.025 per cent Methylene Blue solution and 2γ of selenium in 10 ml solution, various amounts of sodium sulfide solution were added and the decoloration time was measured. As shown in Fig. 1, the decoloration velocity was constant for the amount of 3~5 ml of sodium sulfide solution at 5°, 15° and 20°C, and so, in the subsequent experiments 3.5 ml of sodium sulfide solution (N/10) was used.

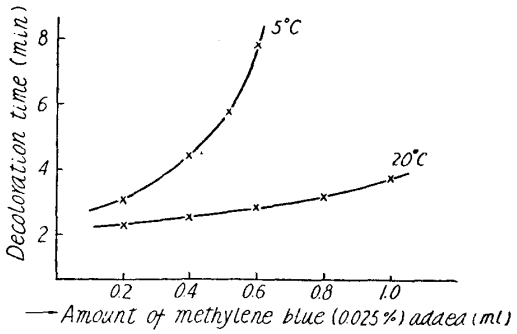

Fig. 1

(ii) Amount of Methylene Blue

To determine the suitable amount of Methylene Blue for the estimation of selenium, the decoloration time was measured, using various amounts of Methylene Blue under the same condition as in the previous experiments. The results are shown in Fig. 2. The decoloration velocity was constant at 20°C in the case of the addition of 0.2~0.6 ml of Methylene Blue solution. The reaction velocity, however, was very slow at 5°C and, especially, in the case of the addition of Methylene Blue over 0.1 ml the constant velocity could not be obtained.

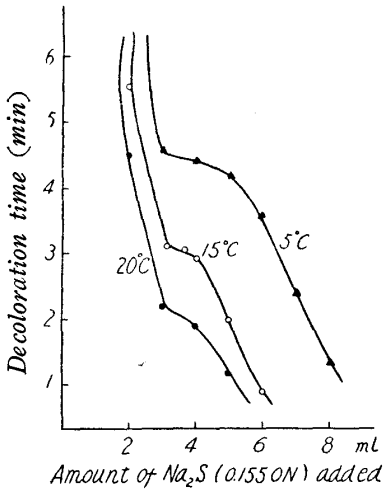

Fig. 2

(iii) Reaction velocity and amount of selenium

Under the condition found in the preliminary work, the reaction velocities for various contents of selenium were measured at 15° and 20°C, and by plotting the reciprocal of time against the amount of selenium, linear relations were obtained as shown in Fig. 3. From the line 1 in Fig. 3, 5~50γ of selenium at 15°C and from the line II, 2~14γ of selenium at 20°C were respectively determined.

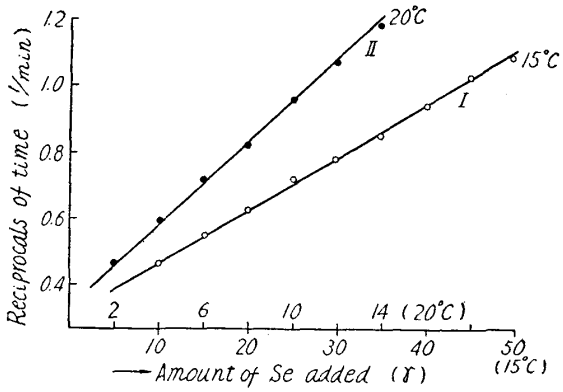

Fig. 3

4. Influences of diverse ions

Under the conditions mentioned above, the influences of some diverse ions were studied at 15°C. As shown in Table 1 most ions could be present till 10γ

without any influence, while Cd^{2+} and Sb^{3+} were present till 60 and 80γ respectively.

Table 1. Influences of diverse ions.

Iron	Metal salt added	Max. amounts of ion permitted
Cu^{2+}	$Cu(NO_3)_2$	10
Pb^{2+}	$Pb(CH_3CO_2)_2$	10
Fe^{2+}	$FeSO_4(NH_4)_2SO_4$	10
Fe^{3+}	$FeCl_3$	10
Ni^{2+}	$NiCl_2$	12
Cd^{2+}	$CdSO_4$	80
Zn^{2+}	$ZnCl_2$	7
Sb^{3+}	$SbCl_3$	60
Co^{2+}	$CoCl_2$	16

Summary

1) Applying the catalytic behavior of selenium to the reaction between sodium sulfide and Methylene Blue, the method for the determination of micro-amount of selenium was studied.

2) Under the condition obtained, 2~14γ of selenium at 20°C and 5~50γ of selenium at 15°C could be determined.

3) Influences of some diverse ions were also investigated.